

**MATHEMATICAL MODEL OF ENERGY
TRANSFERRED IN A SINGLE BALL MOTION IN
AGITATION MODE**

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**MATHEMATICAL MODEL OF ENERGY TRANSFERRED IN A SINGLE
BALL MOTION IN AGITATION MODE**

by

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LIST OF SYMBOLS

a_p	Piston acceleration
a_v	Vial acceleration
a_b	Ball acceleration
c	Connecting rod length
D_b	Ball diameter
e	Coefficient of restitution
e_l	Coefficient of restitution at specific work materials thickness
E_{bb}	Energy loss due to ball bouncing in the collision without the present of work material
E_{bb_l}	Energy loss due to ball bouncing in the collision with the present of work material
E_f	Energy loss due to friction
E_{f+s}	Sum of energy loss due to friction and drag
E_i	Impact energy generated in the collision without the present of work material
E_{il}	Impact energy generated in the collision with the present of work material
E_k	Kinetic energy
E_s	Energy loss due to drag
E_u	Potential energy
g	Gravity acceleration
h	Ball falling height

..continued

h_b	Ball height at rest (on bottom surface of the vial)
h_l	Ball height at departure point
h_{bd}	Ball height upon departure (trajectories)
h_d	Ball height at free falling phase
h_{peak}	Maximum ball height (bouncing)
m	Ball mass
r	Crank radius
s_p	Piston displacement
s_v	Vial displacement
t	time
t_p	Work materials thickness
t_{pc}	Critical work materials thickness
v	Velocity
v_p	Piston velocity
v_v	Vial velocity
v_b	Ball velocity at rest (on bottom surface of the vial)
v_{bd}	Ball velocity upon departure (trajectories)
v_d	Ball velocity at free falling phase

Greek Notations

θ	Crank angle
ω	Angular velocity
μ	Coefficient of friction
ρ_g	Density of work materials

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MODEL MATEMATIK BAGI PERPINDAHAN TENAGA DI DALAM PERGERAKAN BEBOLA TUNGGAL PADA MOD GONCANGAN

ABSTRAK

Proses pengecilan saiz partikel menggunakan teknik pengisaran mekanikal adalah satu proses yang melibatkan penggunaan tenaga yang tinggi tetapi tidak berkesan. Secara umumnya, hanya 1 hingga 5 peratus daripada tenaga yang dibekalkan, digunakan untuk pengecilan saiz partikel manakala selebihnya hilang terutamanya dalam bentuk haba. Memandangkan tenaga merupakan penyumbang utama kepada kos pembuatan, banyak usaha telah dilakukan untuk memahami mekanisme pemecahan partikel di dalam proses pengecilan saiz partikel bagi meningkatkan kecekapan tenaga. Di industri, ketidakberkesanan penggunaan tenaga tidak dapat dielakkan dan terpaksa diterima pakai dengan melanjutkan tempoh pemprosesan. Bagaimanapun, peningkatan tempoh pemprosesan akan mengakibatkan kebarangkalian untuk berlakunya pencemaran ke atas bahan kerja yang dikisar meningkat. Dalam kajian ini, pergerakan balang pengisar, bebola dan bahan kisar di dalam mesin pengisar diselidik bagi memahami mekanisme perlanggaran yang berlaku di antara mereka. Perlanggaran di antara balang pengisar, bebola dan bahan kisar merupakan fenomena yang penting kerana tenaga kinetik dari bebola pengisar akan dipindahkan kepada bahan kisar untuk proses pengecilan saiz. Oleh itu, satu teori permodelan pergerakan balang pengisar, bebola dan bahan pengisar perlu dibangunkan. Untuk mengurangkan komplikasi dalam kejadian perlanggaran oleh sekumpulan bebola yang kebiasaannya diamalkan di industri, pendekatan bebola tunggal yang bergerak pada mod goncangan digunakan. Dengan menggunakan pendekatan tersebut, kajian simulasi telah dijalankan untuk mengenalpasti sama ada tenaga hentaman yang dihasilkan mencukupi untuk membolehkan berlakunya

pecahan di dalam proses pengecilan saiz. Seterusnya, model perpindahan tenaga dari bebola pengisar kepada bahan kisar dibentuk. Pembentukan model ini adalah perlu untuk menilai tenaga sebenar yang digunakan dalam pengecilan saiz partikel. Pembangunan model ini adalah berdasarkan kepada prinsip keabadian tenaga dimana tenaga yang hilang dalam bentuk lesapan geseran dan lantunan bebola diambilkira. Model ini kemudiannya ditentusahkan melalui ujian jatuh-bebas menggunakan bebola pengisar. Ujian ini memberikan senario yang hampir menyerupai perlanggaran terus yang berlaku diantara bebola pengisar, bahan kisar dan permukaan dalaman balang pengisar. Kajian simulasi ke atas model pergerakan balang pengisar, bebola dan bahan kisar mendapati bahawa tenaga hentaman yang dihasilkan ketika perlanggaran di antara bebola pengisar dan permukaan dalaman balang pengisar adalah lebih tinggi jika dibandingkan dengan tenaga impak yang dihasilkan pada mesin pengisar yang lain yang bergerak dalam mod yang sama seperti mesin pengisar getar. Manakala, bagi kajian simulasi keatas model perpindahan tenaga dari bebola pengisar kepada bahan kisar menunjukkan bahawa jumlah tenaga hentaman yang dipindahkan kepada bahan kisar didalam proses pengecilan saiz amat bergantung kepada ketebalan bahan kisar, ketinggian jatuhnya bebola dan berat bebola. Keputusan yang diperolehi dari model teori dan kajian ujikaji adalah setara dimana kedua-duanya memberikan corak keputusan yang sama. Kesimpulannya, tenaga hentaman yang tinggi, yang mencukupi untuk pemecahan bahan dapat dihasilkan dengan menggunakan pendekatan bebola tunggal yang bergerak pada mod guncangan. Jumlah tenaga hentaman yang dipindahkan kepada bahan kisar untuk pengecilan saiz adalah bergantung kepada ketebalan bahan kisar yang terperangkap di antara bebola dan permukaan dalaman balang kisar dan tenaga kinetik bebola. Keputusannya yang diperolehi dapat memberikan pemahaman yang

lebih jelas tentang mekanisme proses pengecilan saiz yang boleh mendorong kepada penambahbaikan rekabentuk mesin pengisar sedia ada supaya kadar pengecilan saiz yang lebih tinggi dapat dihasilkan dengan penggunaan tenaga yang lebih rendah.

MATHEMATICAL MODEL OF ENERGY TRANSFERRED IN A SINGLE BALL MOTION IN AGITATION MODE

ABSTRACT

Particle size reduction process using mechanical milling method is an inefficient energy intensive process. Typically, only 1 to 5 percents of the energy supplied to the process are utilized for the size reduction of the particle, and the remaining energy is dissipated mainly into heat. Since energy is one of the predominant operating costs, a lot of efforts have been expanded to understand the mechanism of particle breakage in size reduction process in order to increase the energy efficiency. In industrial practices, this inefficiency is inevitably accepted by allowing longer processing time. However, the longer the processing time, the higher the probability of contamination in the milled work materials will be. In this work, the motion of the vial, the balls and the work materials in the milling mill was studied in order to understand the mechanism of collisions between them. This is considered as the important event where the kinetic energy of the milling ball is being transferred to the work materials for the size reduction process. Thus, a theoretical model of the motion of the vial, the balls and work materials should be developed. To minimize the complexity of the collision event caused by multi ball commonly used in industry, a single ball approach in agitation motion mode was used. Using this arrangement, simulation was performed to examine whether the impact energy generated would be sufficient for work material breakage in the size reduction process. The next step taken was to model the transfer of energy from the milling media to the work material. This is required in order to evaluate the actual energy used in particle size reduction. The model developed is based on the principle of energy conversion, where the energy lost in terms of frictional dissipation and ball bouncing was considered. The model was then validated by free falling test using a

milling ball. This is the closest resemblance of direct collision between milling ball, work material, and the internal surface of the vial. The simulation results on the model of motion of the vial, ball and work materials show that the impact energy generated during the collision between the milling ball and internal surface of the vial was higher as compared to impact energy generated in other type of mills in this mode of motion, such as vibratory mill. Whereas, the simulation of the model of impact energy transferred to work materials showed that the amount of impact energy transferred to the work material for size reduction process was strongly dependent on the thickness of the work materials, height of falling of the ball, and the mass of the ball. The results obtained from the theoretical model and experimental works were in good agreement where both show a similar trend. Thus, it could be concluded that high impact energy, sufficient for material breakage, could be generated by using the single ball approach in agitation motion mode. The amount of impact energy transferred to the work materials for size reduction is the function of the thickness of work materials trapped between the ball and the internal surface of the vial, and the kinetic energy of the ball. The findings would provide a better understanding of the mechanism of size reduction process which could lead to the improvement of existing design of milling mill, where the higher level of size reduction rate could be achieved using lower energy consumption.

CHAPTER 1

INTRODUCTION

1.0 Introduction

Mechanical milling (MM), which is also known as mechanical grinding, is a process to reduce the physical size of solid particulates from coarse to its micro-structural level by means of mechanical impact. It is a simple processing technique that is now commonly employed in the production of fine or ultra-fine powder of metals and ceramics. Mechanical milling has been traditionally used in powder metallurgy and ceramics for the production of fine powders. In this process, the work materials are subjected to high collision energy in a high-energy ball mill such as planetary, tumbling and vibratory ball mills. Solid particulates, which present between the colliding bodies during collision, are plastically deformed and broken under high impact energy (Dutta and Pradhan, 2002).

The MM process is largely determined by operation conditions such as the number and size of the milling balls, types of milling mill, properties and characteristic of work materials, ball/powder weight and volume ratios, milling velocities etc (Feng et al., 2003). Quantifying and controlling various operation variables for optimum milling process, especially dynamic variables such as rotational speed or agitation frequency of the vial and revolution speed of the disc, are difficult tasks. Regardless on those parameters setting, the main intention is to create effective relative motion of vial, balls and work materials especially balls motion that should be given chances to accelerate at

a sufficient length of travel in order to produce high velocity to generate high impact energy required for fine fragmentation of particulates. However, the amount of work particulates being present between the colliding bodies is another important consideration. The larger the amount of work particulates presents between colliding bodies, the higher the production rate. Thus, better understanding of kinetics of motion of the vial, balls and the milled work materials will provide the necessary knowledge on the mechanism of collision, which is known as a primary transfer event by which energy from the milling balls are transferred to the work materials (Magini et al., 1998). Understanding on the mechanism of motion in various types of mill would help in identifying and designing effective relative motion of vial, balls and work materials with high collision intensity and frequency.

The effectiveness of MM process is dependent upon the intensity and frequency of collisions, where high impact energy could be transferred to the work materials. The impact energy which is generated during the collision is the source of deformation and fracture leading to the breakage of the work materials into smaller sizes. The higher the impact energy, the higher the number of fragments produced, the smaller the size of each fragment (Wu et al., 2004). However, if the impact energy is low, the impact stress generated in the particle of work materials may not be sufficient to cause fragmentation. To obtain optimum refinement amount of work materials, it is important to ensure that each collision produces high impact energy and at the same time, the amount of work materials being present between the colliding bodies is at maximum.

From the energy point of view, MM process typically uses only 1 – 5% of the supplied energy (Clearly, 1998), where most of energy is dissipated into non-particle-breakage process. Thus, MM process usually requires long processing time. To increase the energy efficiency in particle-breakage process, basic understanding on the use of impact energy for particle-breakage should be well understood.

Ideally, kinetic energy of milling ball should be completely transferred to work materials for particles size reduction during collision. In real application however, most of the kinetic energy of the ball is dissipated mainly to frictional heat. Thus, how energy is transferred to the work materials during a collision should also be understood in order to improve the energy usage in MM process. A free falling test using milling ball, which is a closest resemblance to mimic a direct collision between ball and inner wall of the vial in MM process could be used. This represents the basic and simple fundamental arrangement to study on the mechanism of collision and how the kinetic energy of the ball is transferred to the work materials. The related factors that influence the energy transfer to the work materials during the collision could also be identified.

1.1 Problem statement

MM is a simple process and influenced by few variables such as rotational speed, revolution speed and filling ratio. However, the process becomes complex for powder of nano size (Fecht, 1995). The influence of the variables on the effectiveness of MM process have been investigated by many researchers and for several variables, the optimized condition have been identified. Although optimization has been achieved

on some of the variables, these findings are unable to completely improve the MM process. The optimized condition on the identified variables may no longer valid if one of other variables such as different types of work materials or different types of milling system is used. The influence among the variables is also far from being understood (Dashtbayazi and Shokuhfar, 2007).

The energy consumption in MM process was also reported very low. Misra (2003) stated that not more than 20% of the energy was utilized in comminuting. Due to inefficiency in energy utilization, MM process is usually performed in a long processing time that ends up at high processing cost. Previous works showed that longer processing time was not only increasing the cost but also contributed to other problems such as contamination on product and high processing temperature (Enayati et al., 2004 and Kwon et al., 2002). The effect on contamination of long milling period have been proven in many experimental works conducted such as Wang et al. (1995). Thus, minimizing the milling time would reduce the contamination in milling process. In addition, milling at longer duration was also reported to increase the milling temperature. It is believed that the temperature rise during milling is mainly due to balls to balls, balls to powder, balls to wall collisions and frictions (Kwon et al., 2002). Excessive plastic deformation which occurs during milling processes is also a possible sources where heat is released to the milling environment and contributing to the rise of the work materials temperature as well. Increasing the temperature of work materials would reduce the fracture characteristic of the work materials and consequently reduces the efficiency of the milling process.

Thus, it is important to keep milling time as short as possible. This could be achieved if the breakage rate of the work materials is maximized. Theoretically, more fracture could be generated at higher impact energy. Since the impact energy is generated during collision, the studies on the motion behavior of the vial and ball which are responsible in the formation of collision, the mechanism of collision, generation of impact energy and energy utilization in size reduction process ultimately may lead to accomplish an effective milling process with minimum period of processing time.

During the collision, the kinetic energy of the colliding bodies is converted to impact energy which is then transferred to the work materials for deformation and fracturing activities. The kinetic energies involved in the collision have a strong influence in determining the amount of impact energy generated. It is understandable that the amount of impact energy generated during the collision is higher if the kinetic energy of the colliding bodies is high.

The motion behavior of the vial, ball and work materials is the main aspect in controlling the collision event. In order to achieve an effective MM process, the motion of the vial, ball and work materials must be able to produce an ideal collision. An ideal collision is where high impact energy is generated and high amount of work materials is positioned between the colliding bodies, especially between milling balls and internal surface of the vial. The amount of impact energy generated during the collision should be high enough and completely transferred to the work materials for size reduction purposes.

Basically, there are two commonly used methods of motion of charge materials inside the moving vial. The first involves the ‘ball thrown’ method which is commonly provided by conventional ball milling and planetary ball milling techniques, whereas the other using agitation method which is commonly provided by vibratory ball milling and shaker milling techniques.

In ball thrown method, high impact energy could be generated during the collision. However, the random motion of the balls and work materials in the vial create high frictions among them and on the internal surface of the vial. Hence, most of the kinetic energy of the ball is dissipated to heat rather than transferred to the work materials for breakage. Moreover, an excessive friction would contribute to wear and tear of the vial and milling ball. In addition, it has been recognized that in ball thrown method, the probability of the work materials to be trapped in between the colliding bodies is low. In some cases, the amount of the work materials trapped between the colliding bodies could be zero. Since no work materials are trapped, the impact energy generated in the collision could not be transferred to the work material. Thus, particle breakage could not occur.

On the other hand, the collision in agitation methods appears to have a very high probability of work materials trapped between the colliding bodies. This arrangement of vial, ball and work materials motion provides higher tendency of the ball to directly hit the work materials which is located at the bottom surface of the vial. The number of collision is also high due to high frequency of the vial which is up to 20 Hz. However,

the impact energy that could be generated is relatively small as compared to ball thrown method. The length of the vial which is relatively short (maximum 50 mm) provides low amplitude of the ball (Chen et al., 2005). As the ball falls and collides with vial, the kinetic energy of the ball is low and hence, low impact energy is generated. It is envisaged that if the ball could be bounce to higher amplitude, high impact energy could be achieved during the collision.

Although several research groups have investigated the motion behavior of the vial and charge materials for various mills, a model of the motion of vial, ball and work materials that could create an ideal collision is still unknown. Considering of designing an efficient motion of vial and ball in relationship with the work materials that could ensure the optimum impact energy is consumed by the work materials for the size reduction purposes may lead to the improvement of existing designs and to the optimization of the MM process.

To obtain an optimized processing condition, the motion of vial and ball in relationship with the work materials should be designed in such a way to ensure high probability of collision, especially collision between ball and internal wall of the vial, could be occurred. Each of the collisions should be able to trap a maximum amount of work materials. The collision should also be able to generate optimum impact energy that could be consumed by the work materials for the size reduction purposes. In addition, the motion of vial and ball should also be capable to produce high collision intensity and frequency. High collision intensity could be achieved in ball thrown

method. However, high collision frequency could be achieved in agitation method. Developing a new approach of the motion of vial, balls and work materials based on ball thrown method with high collision frequency may be complicated due to its nature of motion which is in random mode. However, considering of development a new approach of the motion of vial, balls and work materials based on agitation method could be possible since the intensity of the collision could be increased by using an agitation system with higher amplitude.

In agitation method, the kinetic energies of the balls and the work materials, which are generated during the oscillatory motion of the vial, will be transformed into impact energy during collision. Since the transformed kinetic energy is a function of mass and the mass of the individual ball is much larger than the mass of the individual particle of the work materials, therefore the ball has higher kinetic energy than the work materials. As a result, the ball will bounce vertically higher than the work materials. Consequently, as the ball moves downward and create a collision with the vial, the work materials will be trapped between the ball and the vial. This is an ideal situation for the mechanical milling process.

The amount of impact energy could also be increased by increasing the agitation amplitude of the vial. The larger the agitation amplitude of the oscillating vial, the higher the bounce of the ball whilst the amplitude of the work materials still remains relatively smaller. Therefore, the impact energy generated between the ball, the trapped work materials, and the vial will be higher.

1.2 Research objectives

Based on the problem statement stated above, the research objectives are as followings:

1. To develop a new model of motion of vial, ball and work materials that improve the collision intensity and frequency. A single ball approach in agitation mode of motion is considered in the development of the model.
2. To develop a model of impact energy transferred to the work materials based on direct collision between ball and inner wall of the container.
3. To identify significant variables such as angular velocities, work materials thickness, falling height and ball mass that increases the impact energy during collision.

1.3 Research scopes

Since the collision is a primary event where the energy is transferred to the work materials for particles size reduction, this work exclusively focused on the mechanism of collision and how the energy from the colliding bodies was transferred to the work materials. The study on the mechanism of collision was limited to direct collision between a single ball and vial which moving in agitation mode. The kinematics equation of the motion of the vial and ball and the equation of impact energy generated during the collision were derived. The motion behavior of the vial and ball, the collision event, the amplitude of the ball bounces and impact energy were examined through simulation works using MATLAB and Microsoft Excel.

Upon the collision, part of the energy will be converted into impact energy while others are dissipated, mostly through friction. The impact energy generated is then transferred to the work materials for particles breakage. A simple free falling test using milling ball, which mimics the collision between ball and vial with some predetermined amount of specified work materials were placed in between, was used to study how the kinetic energy of the ball was transferred to the work materials. Only energy dissipation in the form of frictional dissipation and bouncing were considered.

The theoretical model of impact energy focusing on basic principle of energy conservation was first developed. Simulation works using MATLAB was performed for various sizes of balls, various falling height and various thickness of work materials. The characteristics of the powder were not considered due to equipment limitation. The simulated models were then verified through experimental works where a dynamometer was used to measure the impact energy generated during the impact.

1.4 Thesis organization

The thesis is presented in six chapters as follows:

Chapter 1 reviews the background of the thesis and presents the problem statement, research objectives and scope of research. It also contains the overview of the topics to be included in the thesis.

Chapter 2 reviews the work in the field of mechanical milling process. Its emphasize is on the motion of the vial, balls and work materials which is the important

elements in generating impact energy during collision event. Various configurations of the motion of vial, ball and work materials are reviewed. Kinematics equations of the motion of the vial and ball in various types of mill and equations in predicting the impact energy generated during the collision are highlighted.

Chapter 3 illustrates the proposed mathematical models and implementation of the model developed. Two models were developed; a model of the motion of vial, ball and work materials and a model of impact energy transferred to the work materials during collision. The model of the motion of vial, ball and work materials were developed based on agitation concept at vertical direction using single milling ball. A model of the energy transferred to the work a material was developed based on law of energy conservation principal for free falling ball, which falls from a specific height onto work materials placed in a container. The first model was implemented by simulating the model to visualize the behavior of the model, whereas for the second model, the implementation was accomplished through simulation, which was then validated through experimental work. The methodology of the simulations and experimental procedure were briefly described.

Chapter 4 presents the results from simulation works of the model on motion of vial, ball and work materials. The analysis of the simulation results cover various aspects including, the motion behavior of the vial and the ball, the amplitude of the ball bouncing and the impact energy predicted during the collision. This chapter also includes the simulation and experimental results obtained from the second model;

model of impact energy transferred to the work materials. The influence of the ball mass, falling height and the thickness of work materials were discussed thoroughly.

Chapter 5 presents the conclusions drawn from the research and recommendations for the future research works.

CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

Particle size reduction process using mechanical milling (MM) method is referred to a solid state size reduction process where work materials in the form of coarse particulates are broken into the ultimate fineness by means of mechanical impact energy absorbed by work materials upon the collisions of milling media which are placed inside a reciprocating vial. Many milling techniques have been so far developed to improve the process. However, the efficiency of MM process is still below satisfactory in terms of energy balance, where the energy consumed to reduce the size of particulates is still very low compared to the energy supplied to perform the milling process itself. Since size reduction process of the particulates utilizes the impact energy absorbed from the collisions of milling media, it is important to understand the motion of the balls, the work materials, and the vial, which are the sources of the generation of impact energy. This chapter reviews the works related to the motions of milling media and charge materials, energy utilization and model of energy transfer in MM process. The fundamental of size reduction process and important process variables are also highlighted.

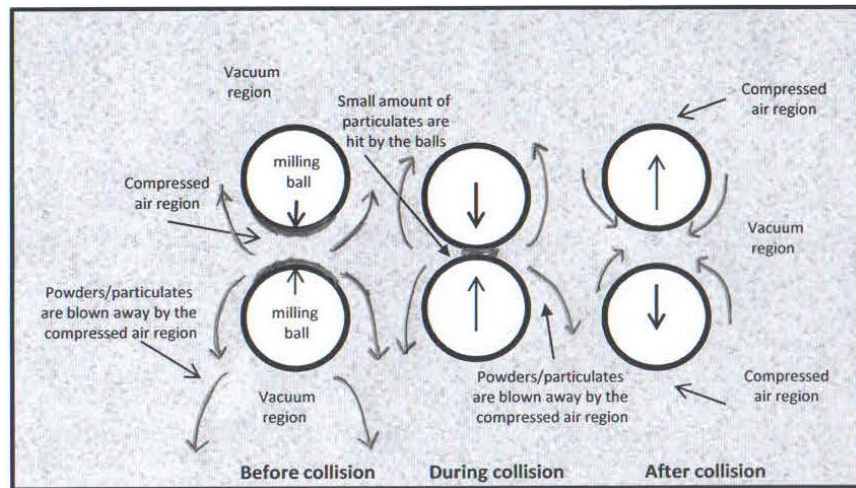
2.1 Mechanical milling principle

The fundamental principle of size reduction in MM process is the energy imparted to the particles during the collision of milling media. During the collision, the kinetic energy of the milling ball as well as the vial is converted into impact

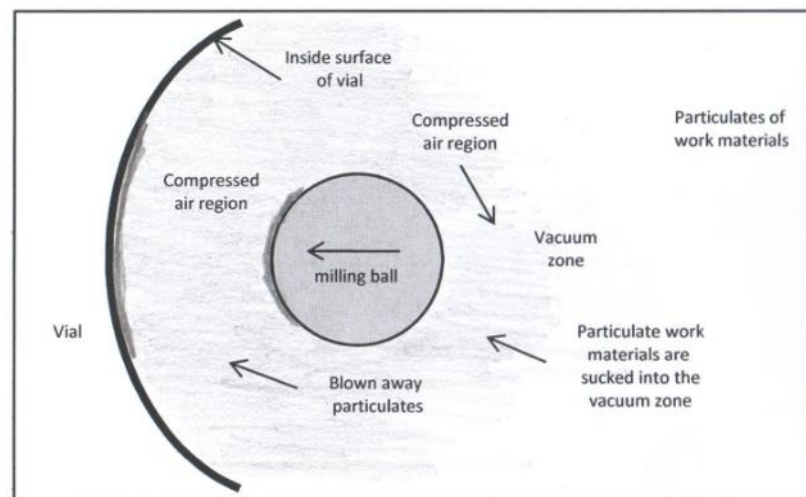
energy. Charge materials for particles fracturing activities then consume the generated impact energy.

The mechanism of collision between two milling balls is illustrated in Figure 2.1.a. When the vial is not vacuumed and the balls are flying inside the vial that is full with particulates of work materials, pocket of compressed air in front of each flying ball projectiles as well as vacuum zone behind the flying ball are generated. Thus, particles, which are present in front of this pocket, are blown away and particles nearby the flying ball are sucked into the vacuum zone behind the ball. Thus, presumably, there are very small amount of particulates, or no particulates at all, present between the two colliding balls. The similar situation occurs in the collision between a milling ball and the inside surface of vial as illustrated in Figure 2.1.b. To avoid the building up of pockets of pressurized air in front of the flying balls as well as the inside surface of moving vial which blow away the particulate work materials, the vial should be made vacuum.

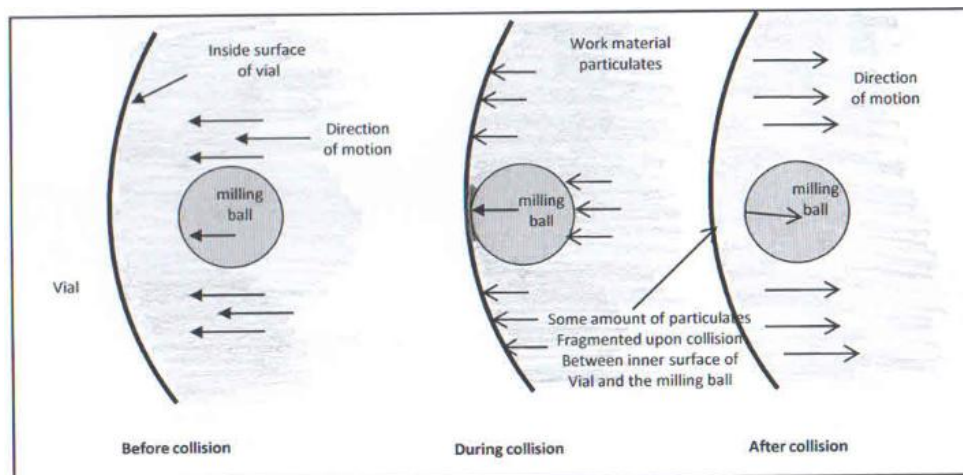
In the vacuumed vial, the milling balls, the particulates of work materials and the inner surface of the vial are moving in such a way that the particulates present in between the flying milling ball and the inner surface of the vial will be crushed. The amount of particulates that are crushed between the ball and the inner surface of the vial is larger than the amount crushed between two milling balls. This is because the colliding area between the ball surface and the inner surface of the vial is larger than the area between two colliding milling balls. Thus, the collision between the ball and the inner surface of the vial is preferable.



(a)



(b)



(c)

Figure 2.1. a) Mechanism of collision between two milling balls inside a non-vacuumed vial b) Mechanism of collision between milling ball and inside surface of vial in a non-vacuumed vial c) Mechanism of collision between milling ball and inside surface of vial in a vacuumed vial.

During the collision, impact force will reduce the porosity of work materials. As the impact progress, the elastic and plastic deformation of the crushed work materials will finally lead to particles fracture, which results in further deformation and fragmentation of the particles.

The general phenomenon during solid-state size reduction is based on fracture mechanics: the nucleation of cracks, followed by crack propagation and fracture, by which new surfaces are formed. For brittle materials, particle fracture was well described by Griffith theory (Rhodes, 1998). According to the theory, the stress, at which crack propagation leading to catastrophic failure (fracture) that occurred in the particles, is approximated by

$$\sigma_F \approx \sqrt{(\gamma E / c)} \quad (2.1)$$

where c is the length of the crack, E is the modulus of elasticity, and γ is the surface energy of the material. The propagation of the cracks is usually occurred very fast with slight plastic deformation. This phenomenon is also known as “brittle fracture”. As the cracks are propagating, stored strain energy is released leading eventually to massive fracture.

In contrary, for ductile materials, the presence of cracks do not play an important role as in the fracture of particles because the excess strain energy is used for the material deformation rather than for crack propagation. Thus, in ductile material, the stress concentration at the top of a crack causes deformation of the material around the cracks tip resulting in a larger tip radius and lower stress concentration. However, the energy required to form a new crack surfaces, γ_s , is generally insignificant compared to the energy required for plastic strain work, γ_p . It

was estimated that γ_s was about $1\text{-}2\text{ Jm}^{-2}$ and γ_p is about 10^2 to 10^3 Jm^{-2} (Vegt, 2007), Thus, due to huge different between γ_s and γ_p , γ_s is usually neglected.

2.2 Process variables

MM process is a complex process and should involve optimization of a number of variables to achieve the desired fineness of the work materials. Typical parameters include type of milling apparatus, milling speed, type of work materials, ball to powder mass ratio (BPMR), ball size and milling time. Many researches have been focused on various types of variables to improve the effectiveness of the process by increasing the impact energy and milling rate. Generally, milling rate is proportional with impact energy. If high impact energy generated, particles breakage and particles deformation of the work materials would absorb high energy, thus, accelerate the milling process.

The following section present detailed discussion on process variables. The effect of the process variables on the performance of the milling process is also highlighted.

2.2.1 Type of work materials

The characteristic of the work materials is one of the variables that determined the effectiveness of the milling process (Ghadiri et al., 2007). It is known that brittle material, such as ceramics is easy to mill whereas, for ductile material, such as metals, the milling process is difficult (Yokoyama and Inoue, 2007). The crack, which is created in ductile material upon the collision, propagates slowly with large amount of plastic deformation. The crack does not extend unless

an increased stress is applied. Thus, to accelerate the milling process, higher impact energy is required.

On the other hand, in brittle material, cracks are propagating rapidly with little or no plastic deformation. The cracks that propagate in a brittle material will continue to grow and increase in magnitude once they are initiated. Hence, less impact energy is required in fracturing the brittle material. Furthermore, brittle fracture usually occurs rapidly and this would accelerate the milling process.

2.2.2 Size of work materials particles

The initial size of work materials plays an important role in determining the effectiveness of the milling process. Basically, long milling time is required to reduce particle from originally in millimeter size to nanometer size. It is known that the plasticity of the work materials increases with the decreasing of the particle size (Vegt, 2007). The author explains transformation from brittle behavior to ductile behavior in relation to particle size theory, which stated that brittle material would show ductile behavior when the particles is smaller than the critical value. When the particle size reaches the critical value, crack propagation is almost impossible. In such situation, high impact energy is required to cause crack propagation on the work materials particles.

Previous work conducted by Shi and Kojavic (2007) shows that the size reduction rate is depending on the starting particles size. Coarse particles tend to be weaker and therefore easier to break rather than smaller particles. Thus, the reduction rate in milling of coarse particle is higher. Gavrilov et al. (1999) have run

the simulation work on a shaker ball mill and the results showed that the milling rate was significantly higher for larger particles. In experimental work conducted by Yang and Shaw (1996), the authors found that the particle size decreases rapidly at the initial stage of milling process and as the particle size becomes smaller, the rate of size reduction decreases slowly. Finally, as the milling continued, the size of the milled particles remains constant.

Similar finding was reported by Ortiz et al. (2008), where at the earlier stage of milling process, the size of milled materials decreases rapidly. The rate of size reduction decreased substantially after 90 min of milling time. After 180 min, the average size of the milled powder remains unchanged at approximately 25 nm. It is predicted that the fracturing of nano particles with the size less than 50 nm, is difficult because the friction force among the particles, which present between the two colliding milling media, counter balances the collision force. Most of the collision energy is consumed by mechanical friction. As a result, the particle size decreases slowly and the milling rate reduces.

2.2.3 Types of milling apparatus

It is known that milling performance is depending on the type of mill used (Suryanarayana, 2001). Generally the milling rate increases with the increasing of the energy of the mill. Most of the common mills used in MM process are planetary mill, tumbling mill, vibratory mill and shaker mill. Usually the faster the mill rotates, the higher would be the energy transferred into the work materials. The schematic diagram of planetary mill, tumbling mill, vibratory mill and shaker mill are shown in Figure 2.2 to Figure 2.4.

In tumbling mill, the milling media (balls) is set in motion by rotating the chamber either on the roller or using a shaft. Owing to the rotation, the ball are lifted and get potential energy which will be transferred into kinetic energy as the ball falls in cascading manner under gravity acceleration. The work materials are dispersed within the milling balls and are stressed by pressure and friction between layers of ball or by impact of falling ball (Kwade and Schwedes, 2007). However at a certain limit of rotational speed, the balls will be pinned to the inner wall of the vial and do not fall down to exert impact energy.

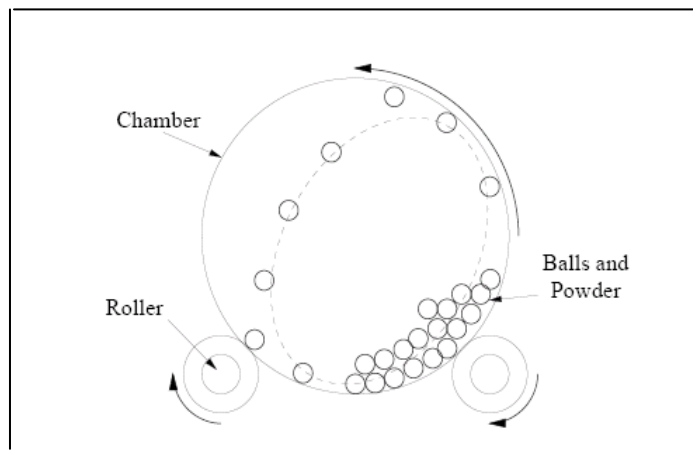


Figure 2.2. Schematic illustration of tumbling mill (Source : Li and Lai, 1998)

In multi-vial planetary ball mill, the vials are mounted on the large rotating disk. The centrifugal force produced by the vials rotating around their own axes and that produced by the rotating support disk both act on the vial contents, consisting of work materials and the milling balls. Since the vials and the supporting disk rotate in opposite directions, the centrifugal forces alternately act in opposite directions. This causes the milling balls to run down the inside wall of the vial, followed by the work

materials and milling balls lifting and traveling freely through the inner chamber of the vial and colliding against the opposing inside wall (Suryanarayana, 2001).

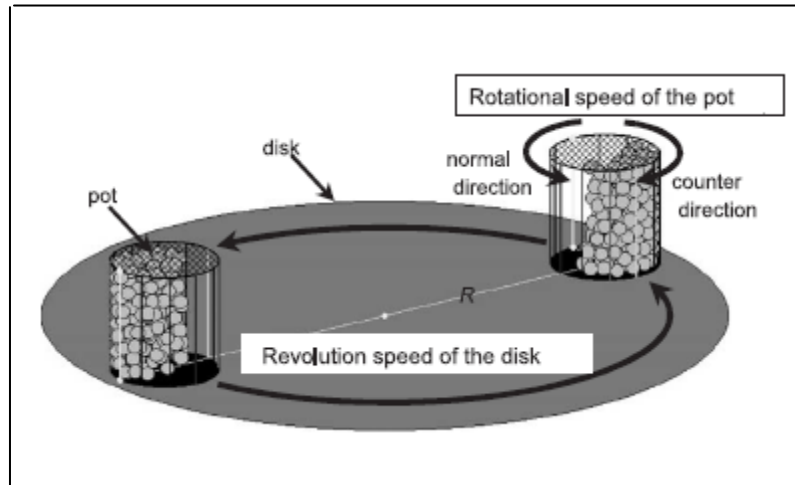


Figure 2.3. Schematic illustration of planetary ball mill (Source : Mio et al. 2004)

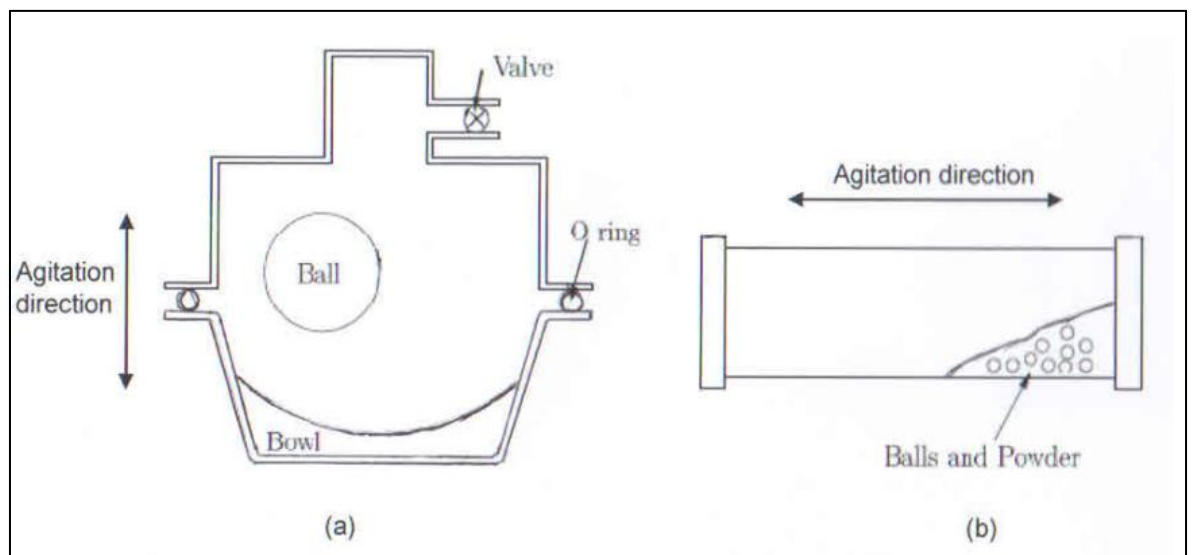


Figure 2.4. Schematic illustration of (a)vibratory mill (b) and shaker mill (Source : Harris, 2002)

In vibratory ball mill (Figure 2.4a), the oscillating movement of the mill agitates the ball and the work materials causing the ball to crush the work materials against the vial wall (Harris, 2002). Whereas in shaker mill (Figure 2.4b), the back-and-forth shaking motion is combined with lateral movements of the ends of the vial, so that the motion of the vial appears to be describing a shape of Figure 8 as it moves (Suryanarayana, 2001). With each swing of the vial, the balls impact against the work materials and the end of the vial, consequently mill the work materials.

Contradicting findings have been published on the effect of different type of mills used for different milling rate. Borner and Eckert (1997) have investigated the effect of energy input by milling iron powders using SPEX shaker mill and Pulverisette planetary mill. The authors determined that the SPEX shaker mill provides the largest impact energy and therefore leads to a fast decrease of grain size to less than 20 nm after four hours of milling time. In other works, Koch (1997) had reported that the milling intensity is higher in shaker mill as compared to tumbling mill.

Schilz et al. (1999) claimed that planetary type mill is much more effective for mechanical alloying than the vibratory mill. Similar results were reported by Szewczak et al. (1999) when Ti-Al powder is milled in vibratory mill and planetary mill. The time required for the alloy to achieve desired crystalline size in high energy planetary mill is about ten times shorter compared to the vibratory mill.

Binczyk et al.(2001) introduced a new type of mill known as electro-magneto-mechanical mill (EMM). The authors have milled Fe-Al-Si alloy powder

and silicon carbide (SiC) powder for 120 seconds and observed that the particle size obtained was equal to particle size that have gone through milling operation for 6 hours using planetary mill. However, no other data was reported to further support the finding.

On the other hand, Mordyuk and Prokopenko (2004) have developed a new type of mill which is known as ultrasonic ball mill. In order to examine the effectiveness of the mill, a mixture of iron-carbon and ferrous nitride powder have been milled. The results showed the meta-stable non-equilibrium solid solution and phase transformation of the powders could be obtained for the much shorter time compared to traditional mechanical alloying in planetary ball mill. However the successful of this mill was still inadequate due to lack of information published.

2.2.4 Rotational and revolution speed

Experimental conducted by Mio et al. (2002) concluded that the milling rate increases with the increasing of revolution radius and revolution speed. The finding was correlated with Yang and Shaw (1996) and Zhang and Liu (2001) findings. However, the incremental trends do not continue indefinitely where at a higher rotational speed, a reduction on the grinding rate is noticed. Palaniandy and Jamil (2009) have also observed similar finding where the crystallite size decreased as the mill speed increased and reached a constant degree at 600 rpm.

The effect of the milling speed on the particle size was also reported by Gheisari et al., (2009). The authors claimed, at low vial rotational speed (150 rpm),

the particles size achieved after 24 hours milling period was relatively high. When the milling speed was increased to 250 rpm, finer particles size was obtained.

In other work, Mio et al. (2004) have conducted a simulation work to determine an optimum revolution and rotation directions. The simulations were running at two conditions;

- i) the rotational direction of the vial was set at the same direction (i.e. normal direction) of the revolution direction of the disc,
- ii) the rotational direction of the vial was set at a counter direction of the revolution direction of the disc.

The simulation results showed that the counter rotational direction of the vial to the disc was effective for improving the milling performance rather than the normal direction. At counter rotational direction, the impact energy generated was much larger than that at the normal rotational direction. The finding was correlated with the result reported by Schilz et al. (1999), who obtained the shortest alloying time for silicon-germanium system at the condition where the rotational direction was in the opposite direction of the disc revolution with the ratio of rotational and revolution speed at 2.5.

2.2.5 Filling ratio

The filling ratio of the vial has a significant effect on the milling process (Burgio et al. 1991). It is necessary that there is enough space for the balls and the work materials particles to move around freely in the milling container. If the filling ratio is large, there is less space for the charge materials to move around, so the

kinetic energy of the charge materials, especially milling balls, is low. In contrary, if the filling ratio is low, there will be bigger space for the charge materials to travel and build up sufficient kinetic energy for the collision. The longer the traveling distance, the higher the kinetic energy could be gained by the charge materials and high impact energy could be achieved during the collision. In simulation works carried out by Cleary (1998), the energy consumption was found to be higher for a mill loaded with filling ratio 40% than mill loaded with filling ratio 50%. Based on Magini-Iasonna energy transfer model, Feng et al. (2008) have designed and calculated the kinetic energy transfer of the ball in vibrating mill at various filling ratio. The results indicated that maximum kinetic energy of the ball was achieved when the vial is 40%–50% full.

2.2.6 Ball to Powder Mass Ratio (BPMR)

Ball to powder mass ratio (BPMR) is another important variable in the milling process and has a significant effect on the time required in achieving a particular size desired. This ratio is to ensure that maximum amount of work materials are in the collision with the maximum amount of milling balls. For example, the time to form an amorphous phase in a Ti-33 at %Al powder mixture in a SPEX mill was 7 hrs at BPMR of 10:1, 2 hrs at BPMR of 50:1 and 1 hrs at BPMR of 100:1 (Suryanarayana, 2001).

Dutta and Pradhan (2002) have milled vanadium oxide sample in a planetary ball mill using different ball-to-powder mass ratio (BPMR) and found that the particle size reduction is much more effective at higher BPMR. This finding was correlated with the Yang and Shaw (1996) results. They have milled a mixture of